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Subtleties in Crystal Structure Solution from Powder Diffraction Data using Simulated Annealing Method

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Beamline(s): X3B1

Introduction: Powder diffraction techniques have traditionally been used for identification and quantification of polycrystalline material and solving simple crystal structures. The information contained in a diffraction pattern obtained from a powder sample has intrinsically less information than that obtained from single crystals as the three-dimensional intensity information is compressed to one dimension. However with the advent of Synchrotron sources and drastic improvement of computation power more complex structures are being solved using powder data. As more complex problems are being tackled using methods such as simulated annealing, systematic grid searches, Monte Carlo and genetic algorithms, it is important to ask how far can we go with these techniques? What if there are subtleties in the crystal structure not built into the model?

Methods and Materials: In order to answer this question we obtained powder diffraction pattern of Ranitidine Hydrochloride(N-(2-{{[5-(Dimethylaminomethyl)-2-furanyl]methlthio}ethyl)-N'-methyl-2-nitro-1,1-ethene-diamine hydrochloride, $C_{13}H_{23}N_4O_3S^+.Cl^-$) . The crystal structure of Ranitidine Hydrochloride has been solved from single crystal data [1] and it is known that the N-ethyl-N'-methyl-2-nitro-1, 1-ehenediamine moiety takes two conformations, were amine NH, etheneCH and nitro O atoms are disordered.

Results: The cell was first indexed using Treor and the space group was identified as P 2 1/n with *a*=18.808Å, *b*=12.981Å, *c*=7.211Å and *β*=95.047°. The structure was solved ab-initio using the simulated annealing program pssp and finally refined using GSAS. Figure 1 shows a Rietveld refinement with R_{wp} = 12.25 and χ^2 = 11.88. Some soft constraints on the bond length and bond angles had to be applied to keep the refinements stable.

Conclusions: From the results of the Rietveld refinement we notice unusually large thermal parameters for atoms N11, N16, C18, O20 and O21, which suggests disorder in this segment of the molecule (figure2). When the structure including disorder was used a significantly better fit was obtained with R_{wp} = 8.39 and χ^2 = 5.56.

References: [1]Toshimasa Ishida, Yasuko In and Masatoshi Inoue, Acta Cryst. **C46**, 1893-1896(1990).

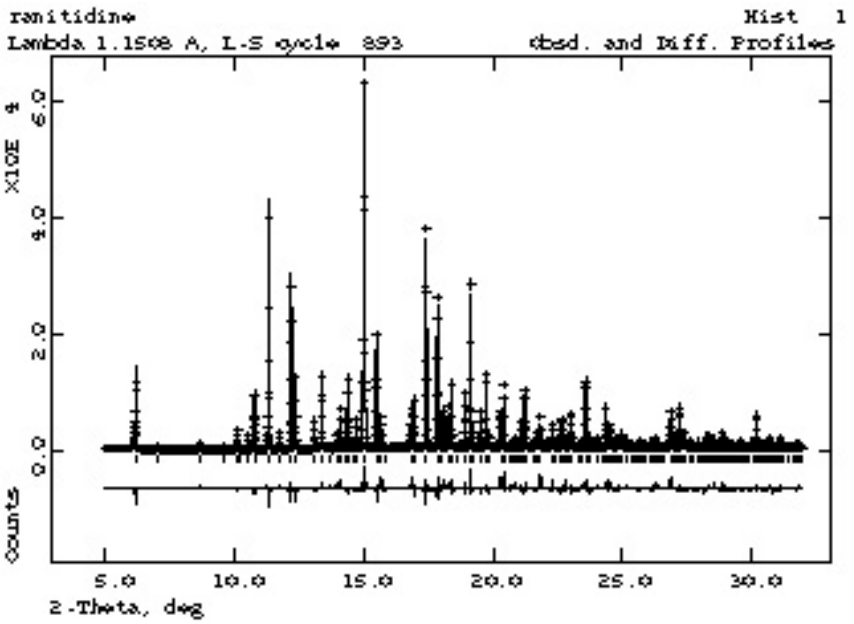

Figure 1: Rietveld refinement of Ranitidine Hydrochloride.

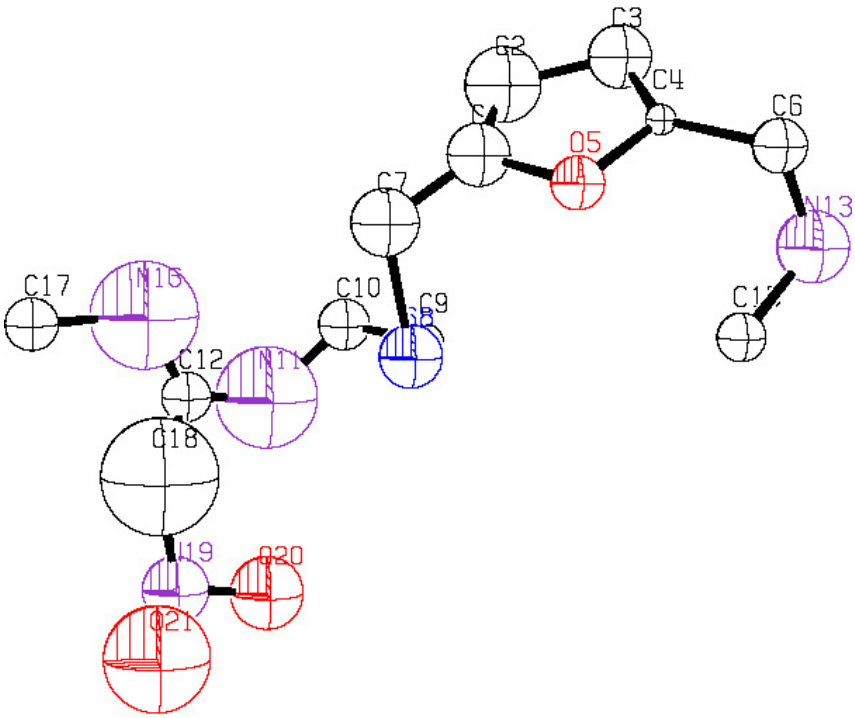

Figure 2: Molecule of Ranitidine showing unusually large thermal paprameters for the disordered atoms.